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NAVORD REPORT 4422

EVALUATION OF COATING TREATMENTS FOR MAGNESIUM ALLOY TUBING

26 OCTOBER 1956

U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

NAVORD Report 4422

EVALUATION OF COATING TREATMENTS FOR MAGNESIUM ALLOY TUBING

Prepared by:

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ABSTRACT: Thirty-two sample sections of Dow Metal FS-1 magnesium alloy tubing, as used for the Submarine Identification Signal (Submerged) XB-7A, were given various coating treatments prior to exposure in a salt spray (fog) test. Three commercial methods for surface treatment of magnesium were considered in this study. Variations of H.A.E. coatings were tested with and without primer coatings. Dow coatings, which had been painted, included Dow No. 1, Dow No. 7, and Dow No. 17 surface treatments. The third method of surface treatment, Iridite 15, was tested painted, unpainted, and partly painted. Maximum exposure time was 240 hours. Failure of coatings necessitated the removal of some tubes at 96 or 192 hours. H.A.E. coatings of .0015 to .002 inch thick, aged, and primer coated provided good protection. Completely painted Dow coatings are all considered acceptable. Iridite 15, when completely painted, is also considered satisfactory. With sufficient protection provided by several of the coating treatments which were studied, cost and processing difficulties may also be used as the basis for selection of the coating treatment for the Signal XB-7A.

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This Report covers the evaluation study of various coating treatments under consideration for use on the Submarine Identification Signal (Submerged) XB-7A. Subject signal is cased in Dow Metal FS-1 magnesium alloy tubing. The purpose of this report is to assemble the information obtained and to make it available to other interested activities. This work was performed under Task 341-366/63012/01040. The observations and conclusions presented are those of the Technical Evaluation Department. Based on the evaluation results, suitable methods for coating treatments will be recommended for use on the subject signal by the Engineering Department.

W. W. WILBOURNE
Captain, USN
Commander

R. E. Hightower

R. E. HIGHTOWER
By direction

CONTENTS

ILLUSTRATIONS

REFERENCES

(a) ASTM Designation: B117-49T
(b) R. M. Burns and W. W. Bradley, "Protective Coatings for Metals", 2nd Edition, Reinhold Publishing Corp., 1955
(c) "New Developments: HAE Finish for Magnesium," Magazine of Magnesium, August, 1953

EVALUATION OF COATING TREATMENTS FOR MAGNESIUM ALLOY TUBING

INTRODUCTION

1. The Submarine Identification Signal (Submerged) XB-7A is designed for use at considerable depths, and is launched by an hydraulic signal ejector from the submerged submarine. This signal is cased in Dow Metal FS-1 magnesium alloy tubing of three inches outside diameter.

2. In conjunction with Task 341-366/63012/01040, the Chemistry Division of the Engineering Department requested various coating treatments be evaluated for use on the Signal XB-7A case. The evaluation program consisted of exposure to salt spray (fog) testing as described in reference (a). This test does not necessarily indicate actual service performance of the coating treatments, but can be applied as an adequate index of performance for comparison of different coating treatments.

3. Three commercial methods for surface treatment of magnesium were tested by using thirty-two sample sections of three inch Dow Metal FS-1 magnesium alloy tubing. Dow coatings were tested with primer coatings or with primer and paint coatings. H.A.E. coatings were tested with and without primer coatings. The third method, Iridite 15, was tested with and without a standard Navy paint system. This standard paint system was also tested without surface treatment of the metal. The coating sequence applied to individual samples is given in Table I.

4. Three types of Dow surface treatment were included in this study. Chapter 16 of reference (b) gives the following information on these coatings. Dow No. 1 is a chrome pickle, consisting of a one-half to two minute dip at room temperature in a sodium dichromate-nitric acid bath, held in air for five seconds, and rinsed in cold, then hot water. This treatment causes an etching action by the solution. Dow No. 7, a dichromate process, is given a one-half to five minute dip in an aqueous hydrofluoric acid solution and a cold water rinse, followed by boiling for thirty minutes in a ten to fifteen percent sodium dichromate solution saturated with magnesium fluoride or calcium fluoride, rinsing and drying. Dow No. 17, which gives an anodized ceramic type coating, is a bath of ammonium acid fluoride, sodium dichromate, and phosphoric acid, applied at 75 to 100 volts, either ac or dc. Treatment time varies with the current, while the current density ranges from 5 to 50 amperes per square foot. Coating

thickness depends on treatment time which ranges from one to thirty minutes.

5. H.A.E coating is an electrolytically applied ceramic coating for magnesium. Reference (c) describes the process as a non-metallic, very hard, adherent finish which is applied by the use of an isolated current source, and is based on the use of 15 amperes per square foot for a treatment time of not more than sixty minutes. A hydrofluoric acid solution post-treatment is followed by aging at 170°F and 80% to 95% R.H. for three hours. Coating thickness varies with the treatment time. Sample tubes used for the tests herein reported were designated as high voltage or low voltage coatings, and aged or unaged. Further details of the treatment process of these tubes was not supplied.

6. Chemically, Iridite 15 is known as a chromate conversion coating. That is, it converts the bare magnesium surface to an entirely new and chemically different surface. This new surface is best described as a complex chromium-chromate. The treatment consists of a chemical dip at 75° to 100°F for one-fourth to two minutes of immersion, a water rinse, and a hot air drying at 160°F to 200°F.

TEST OBSERVATIONS AND RESULTS

7. Observations were made daily during the exposure of the tubes. Coating penetration by the salt fog was detected by the formation of magnesium hydroxide. A pattern of lengthwise stripes was noted on some of the tubes with H.A.E. coatings. This appears to be associated with the surface condition resulting from extrusion of the magnesium alloy since the untreated tubes have a lined appearance in the longitudinal direction. The condition of H.A.E. treated tubes as removed from exposure is shown in Figures 1 through 4. Tubes given Dow treatments are shown in Figures 5 and 6, and those with Iridite 15 are shown in Figures 7 through 9. Tubes which had not been given a surface treatment are shown in Figure 9.

H.A.E. Surface Treatments

8. Tube No. 15 had a high voltage H.A.E. coating, .0002 inch thick, and had been aged. This system showed the greatest coating penetration by salt spray (fog) testing. Heavy lengthwise markings were noted within 23 hours of salt fog exposure. Figure 1 shows the condition of the tube when removed from the test after 96 hours of exposure. After photographing the unwashed tube, warm water and a soft brush were used to remove the magnesium hydroxide and salt prior to

rephotographing. The washed tube indicates the large amount of surface pitting. Two coats of zinc chromate primer in addition to the above H.A.E. aged coating (tube No. 16) provided protection for less than 48 hours. Figure 3 indicates the extent of coating penetration on tube No. 16 by 192 hours of exposure.

9. Low voltage H.A.E. was applied .0003 inch thick on four tubes. Tubes No. 12 and 14 were aged, tubes No. 11 and 13 were not. Two coats of zinc chromate primer were applied to tubes No. 13 and 14. Some breakdown was noted on tubes No. 11 and 12 by 23 hours of exposure. Figure 2 shows the condition of these tubes after removal from 96 hours of salt spray (fog) testing. Aging of tube No. 12 did not provide much more protection than the unaged coating on tube No. 11. Comparison of tubes No. 13 and 14, which had a primer coating, shows a greater difference between the aged and unaged tubes. Blisters were noted on the unaged tube No. 13 by 165 hours of exposure, and peeling had occurred as noted in Figure 4 by 240 hours. The aged sample, No. 14 showed breakdown in a few dots on the inside of the tube by 192 hours, and by 240 hours the lower edge had failed (Figure 4) but no blistering of the coating had occurred.

10. High voltage H.A.E. coating was increased to a thickness of .001 inch on tubes No. 17 and 18, and two coats of zinc chromate primer were applied over the aged coating on tube No. 18. Breakdown was noted on tube No. 17 by 23 hours. The tube was removed after 96 hours of exposure. Figure 2 illustrates the extent of breakdown on the inside as well as the outside of the tube. The two coats of primer provided some protection for the outside of the tube, but did not protect the inside of the tube as shown in Figure 4.

11. When high voltage H.A.E. coatings were applied .0015 inch thick and aged on tubes No. 19 and 20, an increase in protection was noted over the thinner applications. Some slight breakdown was noted on No. 19 after 48 hours. After removal at 192 hours of exposure, examination indicated greater breakdown on the inside of the tube than on the outside (Figure 3). Tube No. 20, which had been given two coats of zinc chromate primer, also showed greater breakdown on the inside of the tube. Figure 4 indicates very little breakdown on the outside other than that noted on the lower edge of the tube.

12. High voltage H.A.E. coating, .002 inch thick, and aged provided the greatest protection of the H.A.E. coatings which were tested. Tube No. 21 showed slight breakdown after 192 hours of exposure (Figure 3). When two coats of zinc chromate primer were applied on tube No. 22, very slight breakdown was noted on the inside of the tube and none on the outside (Figure 4).

Dow Surface Treatments

13. Duplicate samples were available of those systems which included Dow surface treatments. Tubes No. 1 and 2, which had two coats of AP 10 primer over Dow No. 1, showed slight breakdown after 165 hours of exposure. Figure 5 shows their condition when removed after 240 hours. Some breakdown was noted on the edges of tubes No. 3, 4, and 5 (Figure 5) which had one coat of black enamel in addition to the treatment given tubes No. 1 and 2.

14. Coating systems which included Dow No. 7 and Dow No. 17 were tested for 240 hours. As indicated by Figure 6, no breakdown was noted on Dow No. 7 tubes. (Tube No. 6 shows an uneven painted surface at the clamp mark obtained during processing.) Most of the breakdown and blisters noted on Dow No. 17 tubes may be attributed to inadequate paint coating at clamp marks obtained during processing. In addition, some breakdown was noted on the edges of tube No. 10.

Iridite 15 Surface Treatment

15. Duplicate samples were available of those systems which included Iridite 15. Two tubes, No. 25 and 26, were painted over the Iridite coating only on the upper half of the tube. The unpainted Iridite 15 coating was heavily marked within 24 hours of exposure, but breakdown was not noted on the painted half of the tubes until 165 hours. The large amount of blistering and chipping of the painted surface by 240 hours (as noted in Figure 7) may be the effect of lifting of the paint film from the underside, or by the corroding magnesium on the unpainted half of the tubes. For this reason, additional Iridite 15 tubes were tested either completely painted or unpainted.

16. Three unpainted tubes with Iridite 15 were heavily marked within 24 hours of exposure. Tubes No. 23 and 24 were removed from testing after 96 hours. Tube No. 27 was photographed after 96 hours and returned to the salt spray test for additional exposure. The additional breakdown noted on tube No. 27 by 240 hours may be compared with 96 hours exposure on tubes No. 23 and 27 in Figure 8.

17. Three tubes with Iridite 15 were given a standard Navy paint system and salt spray tested for 240 hours. Tube No. 28 blistered only on the top edge as noted in Figure 9, and tubes No. 30 and 32 did not show any breakdown.

Painted Without Surface Treatment

18. A standard Navy paint system was tested on tubes which had not been given the Iridite 15 surface treatment. These tubes, No. 29 and 31, were exposed for 240 hours. As shown by Figure 9, the paint coating blistered on the top edges and lifted from the tubing surface at the edge. Otherwise, the surface of the tubes appeared to be unchanged. Comparison of these results with those of painted tubes with a chemical film surface treatment would suggest no benefit from the surface treatment. However, according to reference (b), paint coatings may be attacked by the basic corrosion products of magnesium, causing underfilm attack which would result in peeling and blistering of the paint coating and loss of paint adhesion over an extended period of time.

CONCLUSIONS

19. The following conclusions are based on the results of salt spray (fog) testing. In general, edge effects are not regarded as sufficient evidence for rejection of coating treatments. Performance under service conditions should be checked to substantiate the laboratory results.

20. Unpainted H.A.E. coatings are not sufficient as a final coating for magnesium alloy tubing. Primer coats over aged .0015 to .002 inch H.A.E. coatings provided good protection as shown by tubes No. 20 and 22. H.A.E. coatings of less thickness may be feasible if given a complete paint system; however, information indicating the minimum thickness required is not available from tests conducted in this study.

21. When given a complete paint system, Dow coatings gave satisfactory protection on all samples. Observations (based on edge effects) made on the samples show the three surface treatments tested to be ranked in preferred order as Dow No. 7, Dow No. 17, and Dow No. 1.

22. Iridite 15, if unpainted, is not suitable as a final coating for magnesium alloy tubing. The application of a standard Navy paint system over the Iridite 15 provided sufficient protection.

23. The standard Navy paint system, tested without a surface treatment of the magnesium alloy tubing, blistered only on the edges of the tubes. Although these tubes appeared to be protected on the surface, further study should be made on the paint adhesion before this system is recommended for use without a surface treatment.

24. With sufficient protection provided by several of the coating treatments which were studied, the cost, processing difficulties, and related subjects may also be used as the basis for selection of the coating treatment for the Submarine Identification Signal (Submerged) XB-7A.

15
UNWASHED

15
WASHED

FIG. I SALT SPRAY (FOG) TEST – 96 HOURS
H.A.E. COATING ON MAGNESIUM TUBE (SEE TABLE I)

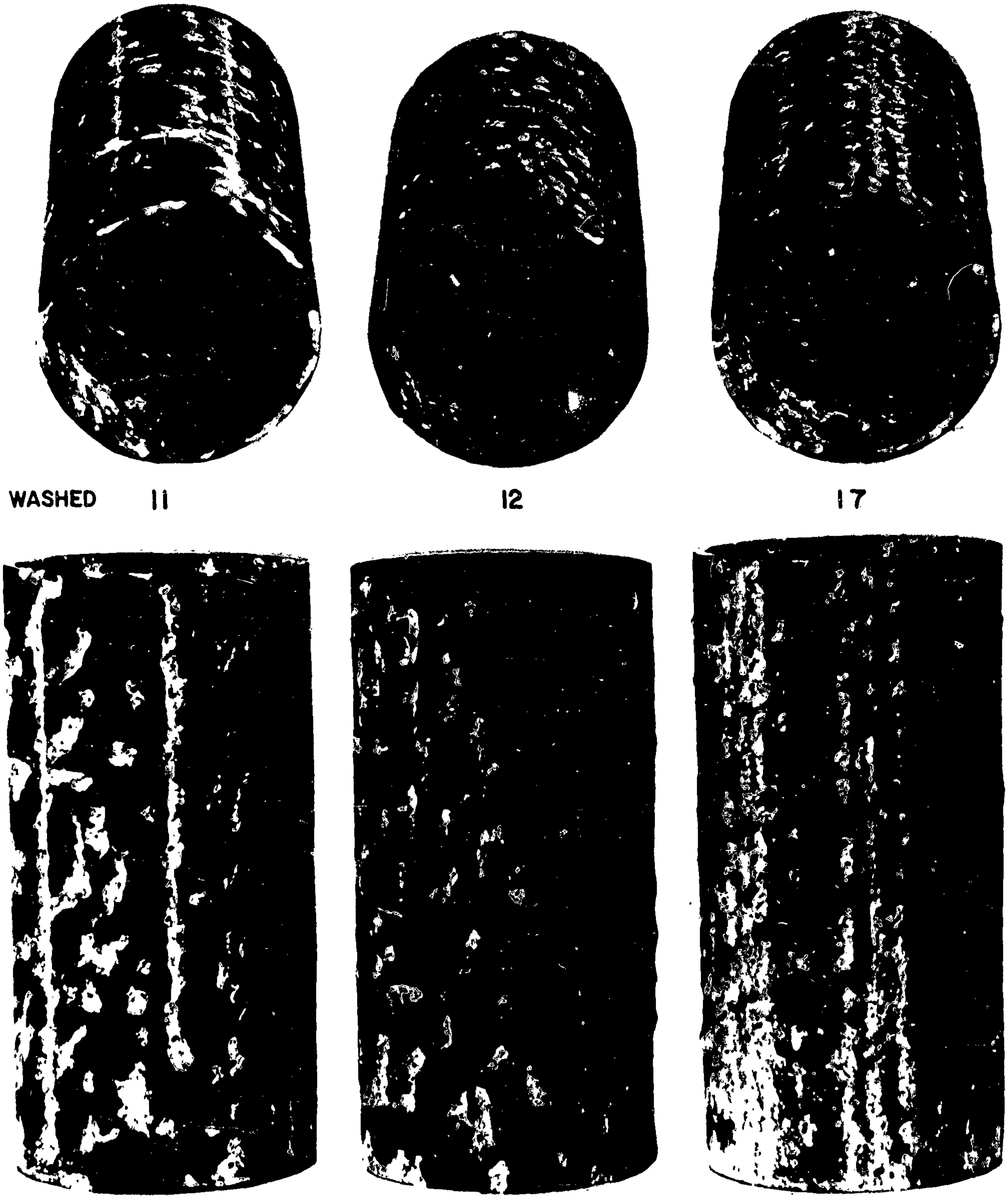


FIG. 2 SALT SPRAY (FOG) TEST – 96 HOURS
VARIOUS H.A.E. COATINGS ON MAGNESIUM TUBES (SEE TABLE I)

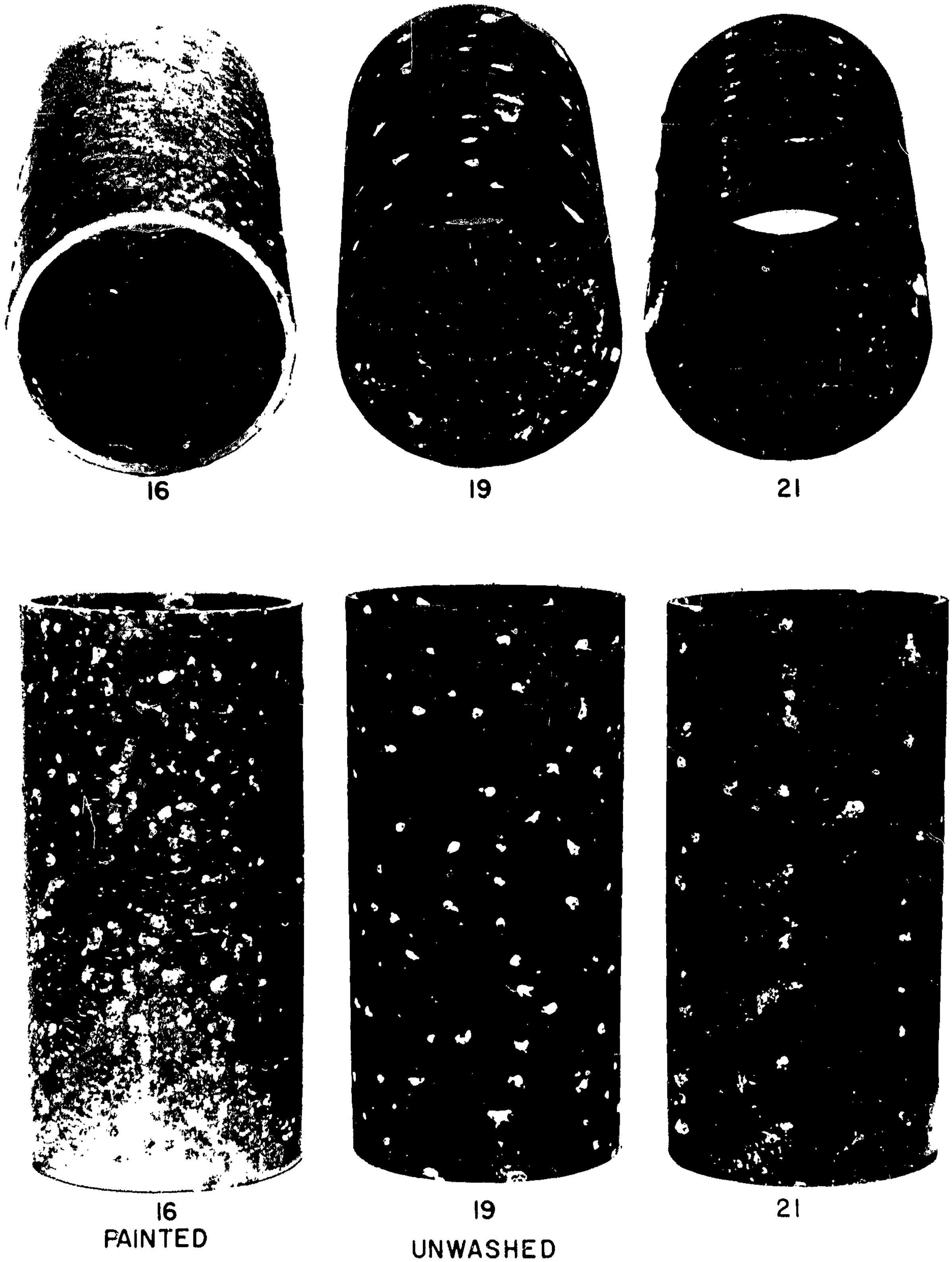
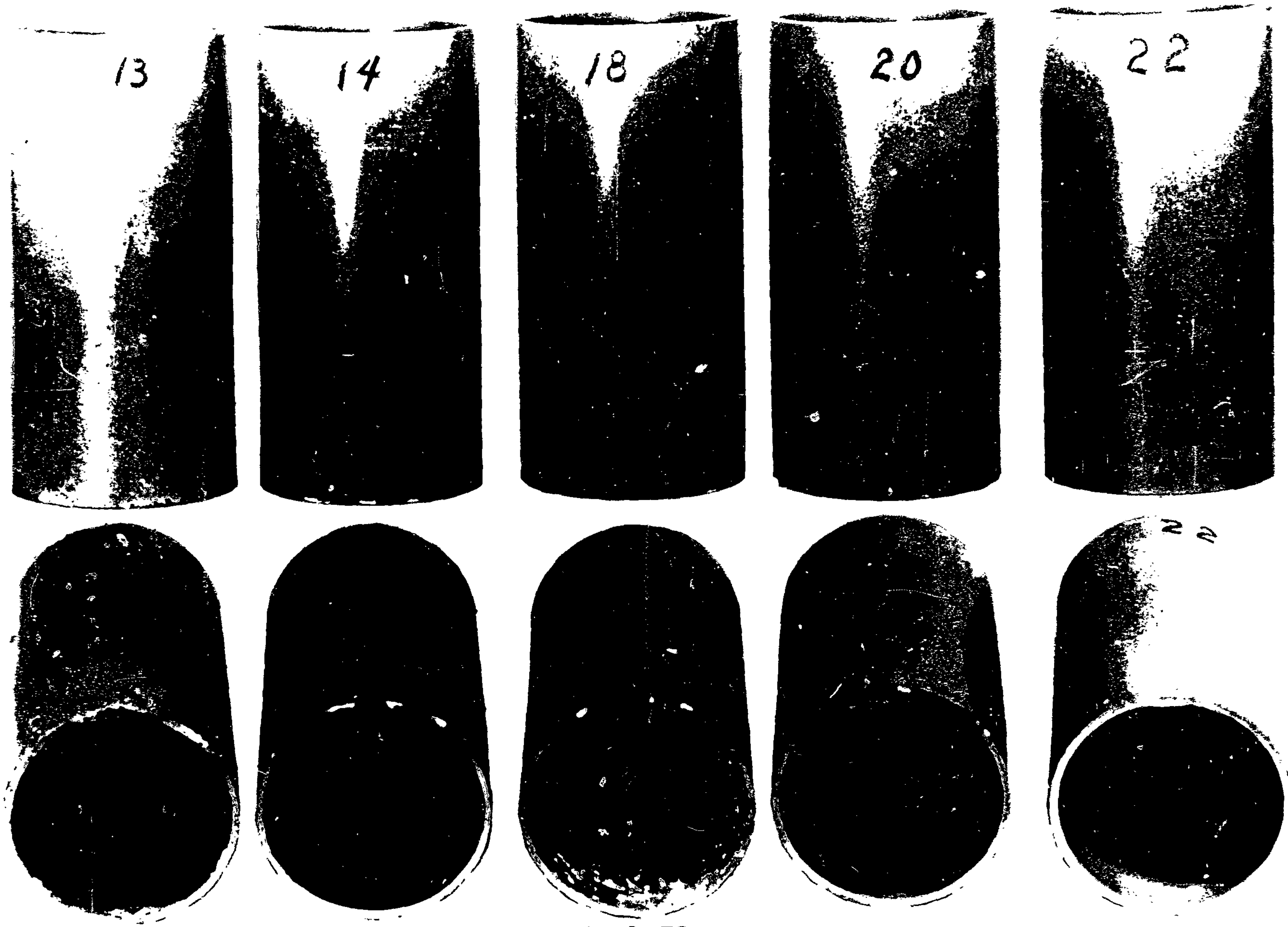
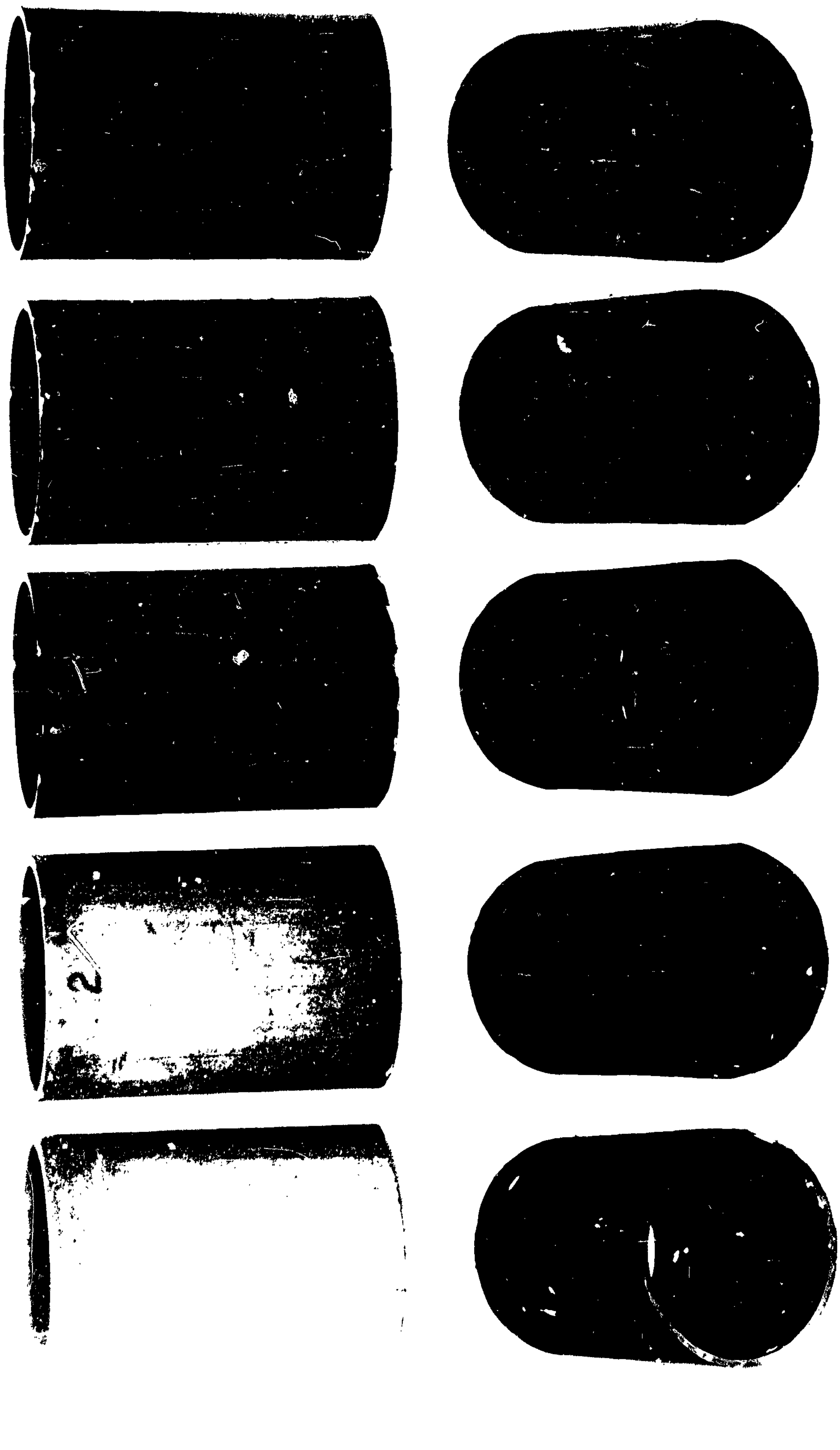


FIG. 3 SALT SPRAY (FOG) TEST - 192 HOURS
VARIOUS H. A. E. COATINGS ON MAGNESIUM TUBES (SEE TABLE I)



WASHED

FIG. 4 SALT SPRAY (FOG) TEST — 240 HOURS
PAINTED VARIOUS H.A.E. COATINGS ON MAGNESIUM TUBES (SEE TABLE I)



WASHED

FIG. 5 SALT SPRAY (FOG) TEST - 240 HOURS
PAINTED DOW No. I COATINGS ON MAGNESIUM TUBES (SEE TABLE I)

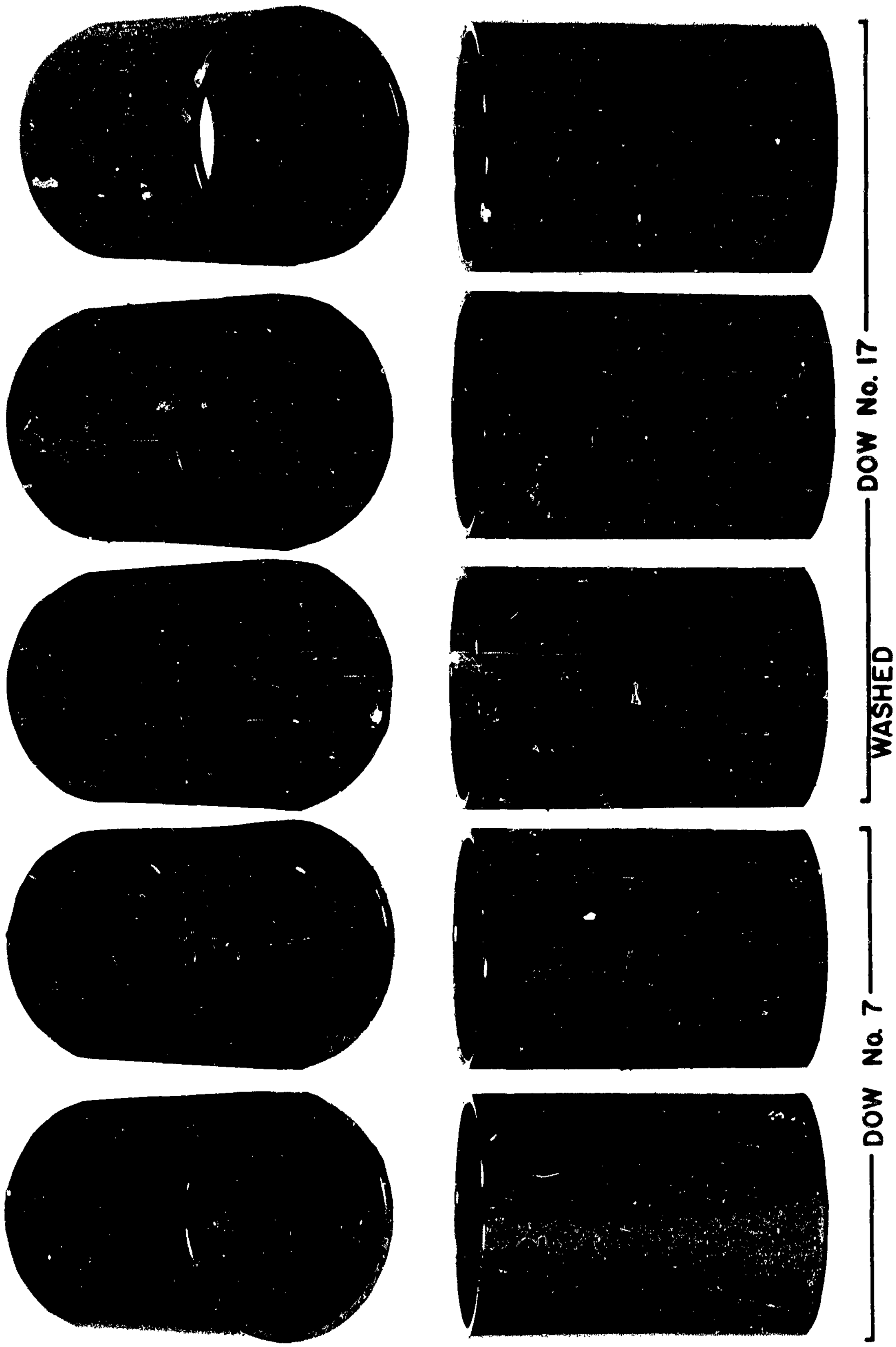


FIG. 6 SALT SPRAY (FOG) TEST–240 HOURS
PAINTED DOW COATINGS ON MAGNESIUM TUBES (SEE TABLE I)

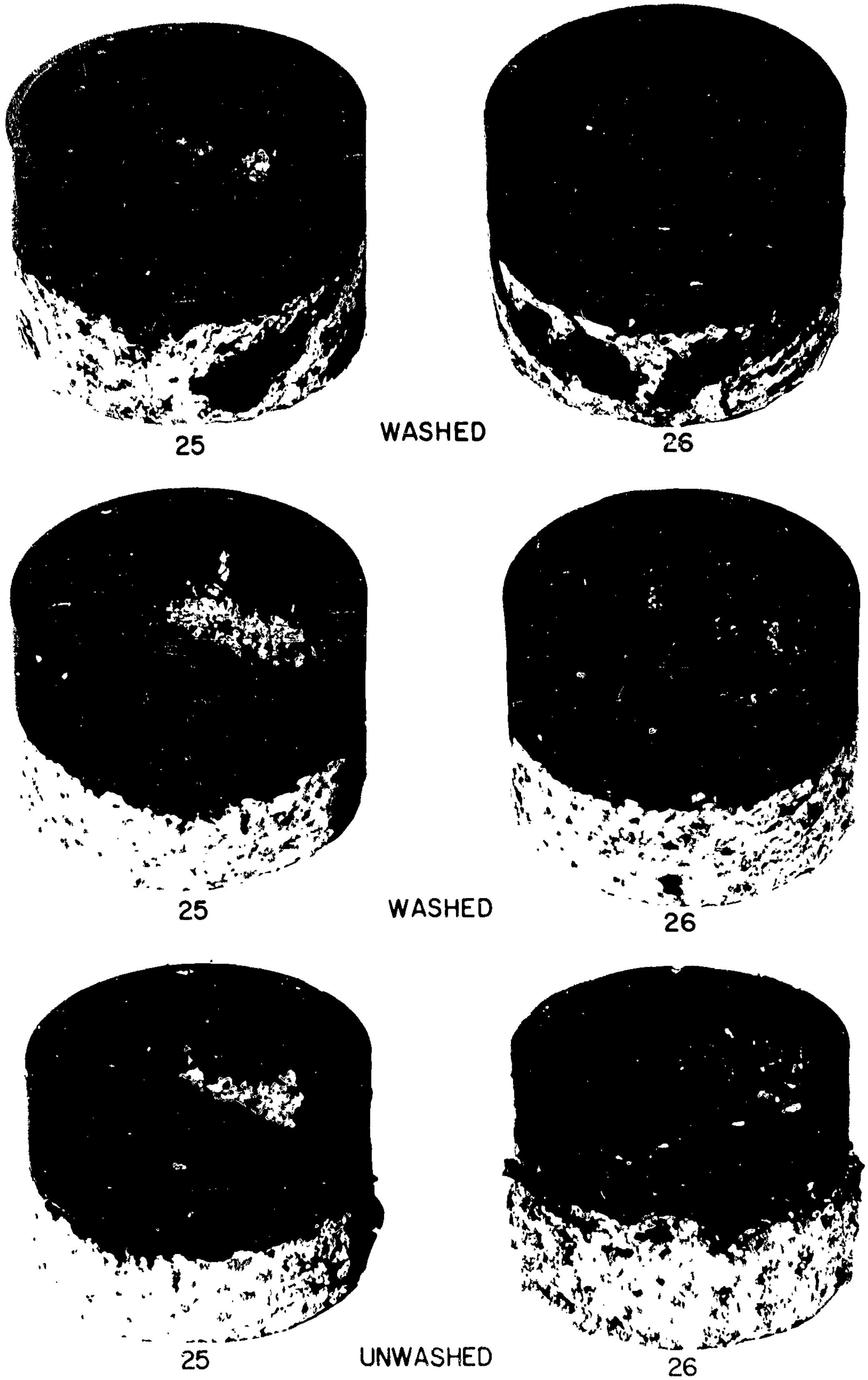
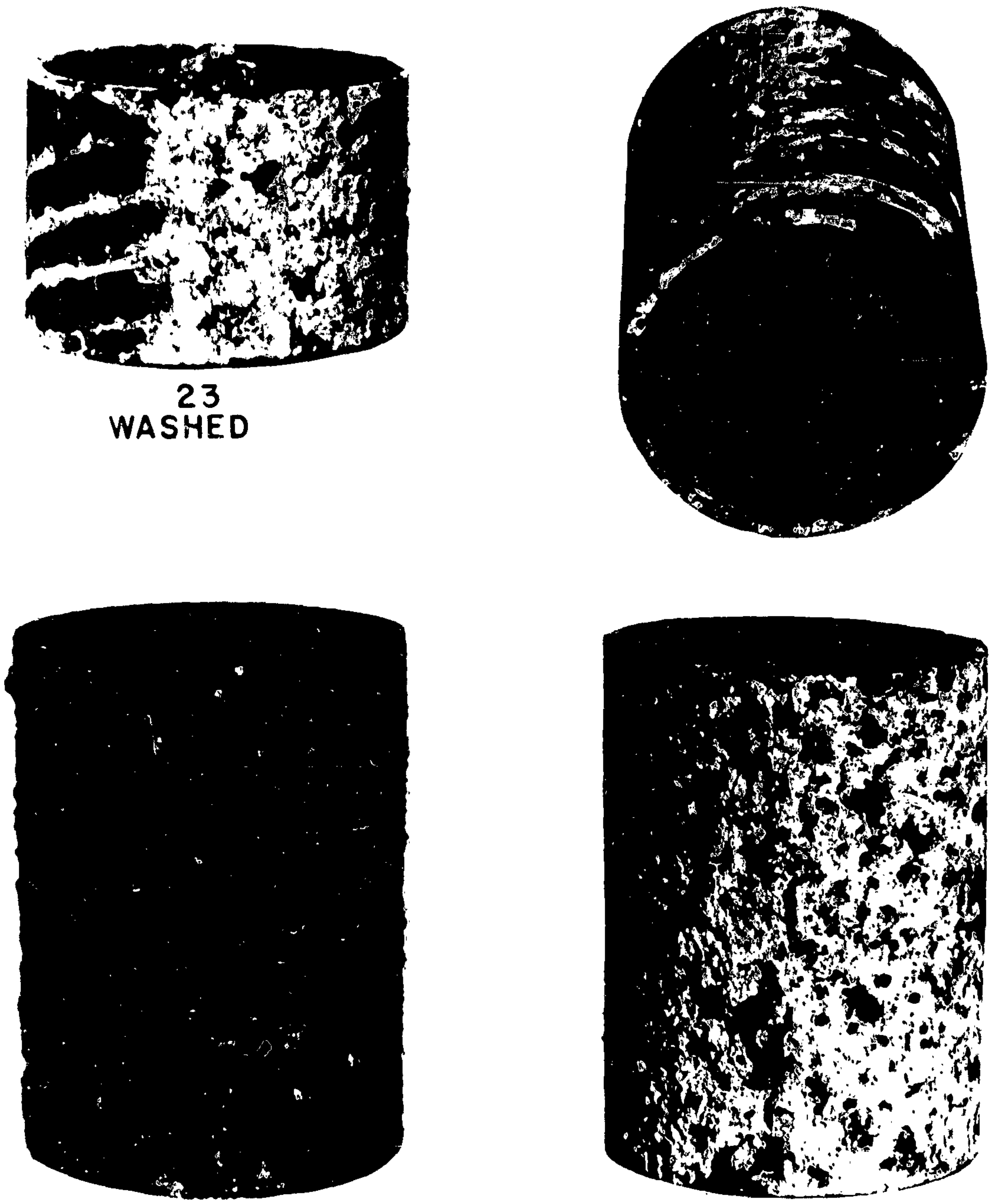


FIG. 7 SALT SPRAY (FOG) TEST- 240 HOURS
IRIDITE 15 COATING (PARTLY PAINTED) ON MAGNESIUM TUBES
(SEE TABLE 1)



23
WASHED

27
WET, UNWASHED
96 HOURS

27
WASHED
240 HOURS

FIG. 8 SALT SPRAY (FOG) TEST
IRIDITE 15 COATING ON MAGNESIUM TUBES

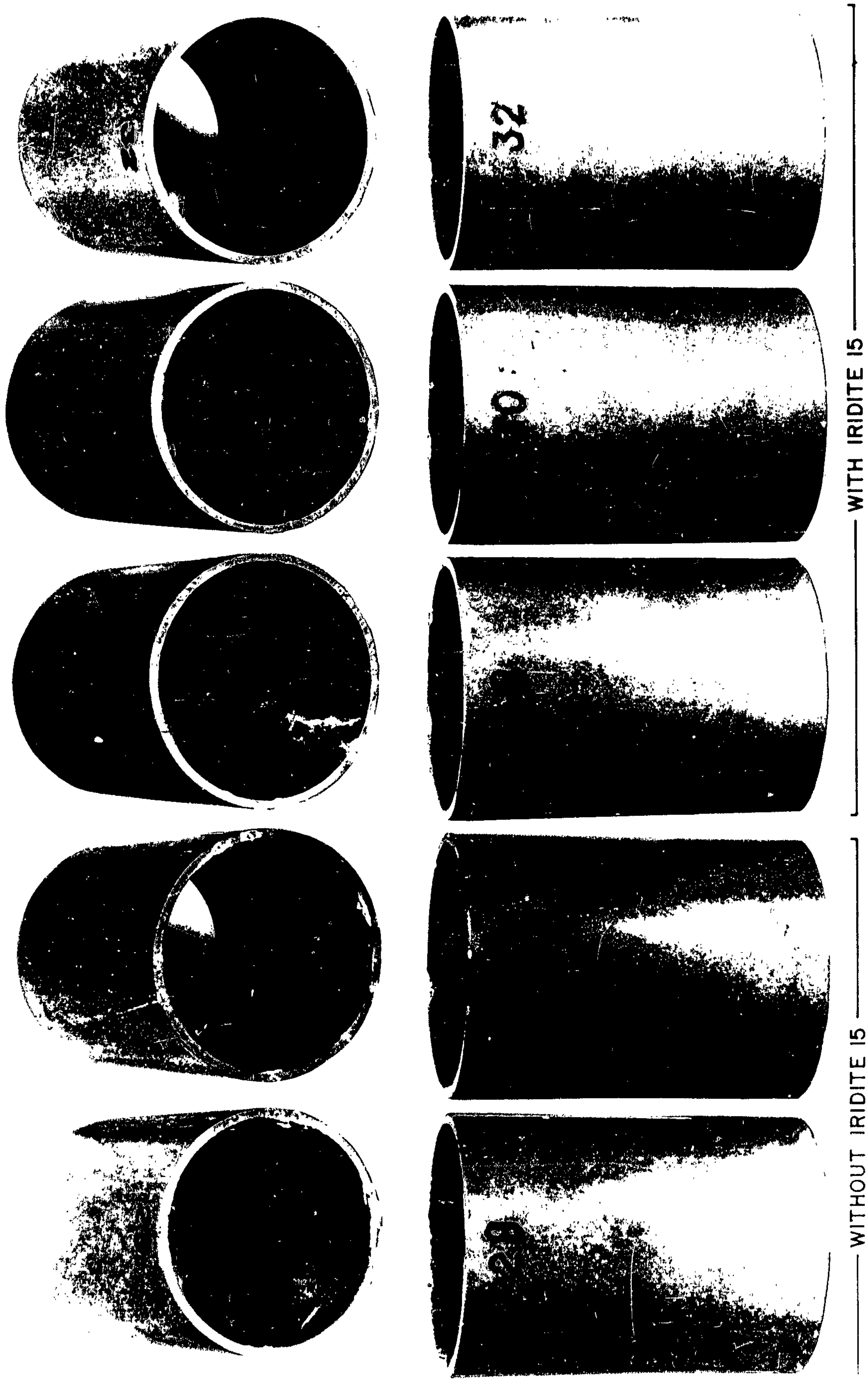


FIG. 9 SALT SPRAY (FOG) TEST — 240 HOURS
PAINT COATINGS ON MAGNESIUM TUBES (SEE TABLE I)

Table I. Coating Treatment of Magnesium Tubes

Tube No.	Length of tubing in inches	Treatment
1,2	4-1/8	Dow No. 1 Two coats AP10 Primer
3,4,5	4-1/8	Dow No. 1 Two coats AP10 Primer One coat Enamel, heat-resisting, glyceryl-phthalate, black, MIL-E-5557
6,7	4-1/8	Dow No. 7 One coat Coating, pretreatment, MIL-C-15328A One coat Primer, paint, vinyl-zinc chromate type, MIL-P-15930A Two thin coats Paint, outside, gray, No. 27 (vinyl-alkyd), MIL-P-15936A
8,9,10	4-18	Dow No. 17 One coat Primer, paint, vinyl-zinc chromate type, MIL-P-15930A Two thin coats Paint, outside, gray, No. 27 (vinyl-alkyd), MIL-P-15936A
11	6	Low voltage H.A.E., .0003 inch thick, no aging
12	6	Low voltage H.A.E., .0003 inch thick, aged
13	6	Low voltage H.A.E., .0003 inch thick, no aging Two coats Primer, zinc chromate MIL-P-6889A
14	6	Low voltage H.A.E., .0003 inch thick, aged Two coats Primer, zinc chromate, MIL-P-6889A
15	6	High voltage H.A.E., .0002 inch thick, aged

Table I. Coating Treatment of Magnesium Tubes

Tube No.	Length of tubing in inches	Treatment
1,2	4-1/8	Dow No. 1 Two coats AP10 Primer
3,4,5	4-1/8	Dow No. 1 Two coats AP10 Primer One coat Enamel, heat-resisting, glyceryl-phthalate, black, MIL-E-5557
6,7	4-1/8	Dow No. 7 One coat Coating, pretreatment, MIL-C-15328A One coat Primer, paint, vinyl-zinc chromate type, MIL-P-15930A Two thin coats Paint, outside, gray, No. 27 (vinyl-alkyd), MIL-P-15936A
8,9,10	4-18	Dow No. 17 One coat Primer, paint, vinyl-zinc chromate type, MIL-P-15930A Two thin coats Paint, outside, gray, No. 27 (vinyl-alkyd), MIL-P-15936A
11	6	Low voltage H.A.E., .0003 inch thick, no aging
12	6	Low voltage H.A.E., .0003 inch thick, aged
13	6	Low voltage H.A.E., .0003 inch thick, no aging Two coats Primer, zinc chromate MIL-P-6889A
14	6	Low voltage H.A.E., .0003 inch thick, aged Two coats Primer, zinc chromate, MIL-P-6889A
15	6	High voltage H.A.E., .0002 inch thick, aged

Table I. (continued)

Tube No.	Length of tubing in inches	Treatment
16	6	High voltage H.A.E., .0002 inch thick, aged Two coats Primer, zinc chromate, MIL-P-6889A
17	6	High voltage H.A.E., .001 inch thick, aged
18	6	High voltage H.A.E., .001 inch thick, aged Two coats Primer, zinc chromate, MIL-P-6889A
19	6	High voltage H.A.E., .0015 inch thick, aged
20	6	High voltage H.A.E., .0015 inch thick, aged Two coats Primer, zinc chromate, MIL-P-6889A
21	6	High voltage H.A.E., .002 inch thick, aged
22	6	High voltage H.A.E., .002 inch thick, aged Two coats Primer, zinc chromate, MIL-P-6889A
23,24	2 - 2-1/8	Iridite 15
25,26	2 - 2-1/8	Lower half: Iridite 15 Top half: Iridite 15, NAVORD OSTD 52, 20th rev., System 22*
27	4	Iridite 15
28,30,32	4	Iridite 15 NAVORD OSTD 52, 20th rev., System 22*
29,31	4	NAVORD OSTD 52, 20th rev., System 22*

Note: * System 22 is: one coat Primer, zinc chromate, MIL-P-6889
two coats, Paint, outside, haze-gray,
No. 27, MIL-P-15130

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